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Technical

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POLYCARBONATE RESIN COMPOSITION
(51) [International Patent Classification, 6th Edition]
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C08G 64/18 NQA
C08K 5/521 KKM
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Hasegawa Koji

Abstract

(57)□□□□

(57) [Abstract]

□□□□

[Problems to be Solved by the Invention]

It is superior in flame resistance, mechanical*thermal property, it offers polycarbonate resin composition which at sametime is superior in external appearance.

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[Means to Solve the Problems]

polycarbonate resin composition* where it consists of polycarbonate resin 1~99 weight% and polycarbonate-organopolysiloxane copolymer 99~1 weight% and content of silicon combines polytetrafluoroethylene 0.2~2 parts by weight which possesses phosphate ester-based compound 0.1~5 parts by weight and the fibril-forming ability and vis-a-vis polycarbonate resin 100 parts by weight under 0.5 weight%, becomes

Claims

□ □ □ □ □ □ □ □ □

[Claim(s)]

□□□□ 10

[Claim 1]

polycarbonate resin composition* where it consists of (A) polycarbonate resin1~99 weight% and (B) polycarbonate-organopolysiloxane copolymer99~1 weight% and the content of silicon combines (C) phosphate ester-based compound0.1~5 parts by weight and polytetrafluoroethylene0.2~2 parts by weight which possesses(D) fibril-forming ability vis-a-vis polycarbonate resin100 parts by weight under 0.5 weight%, becomes

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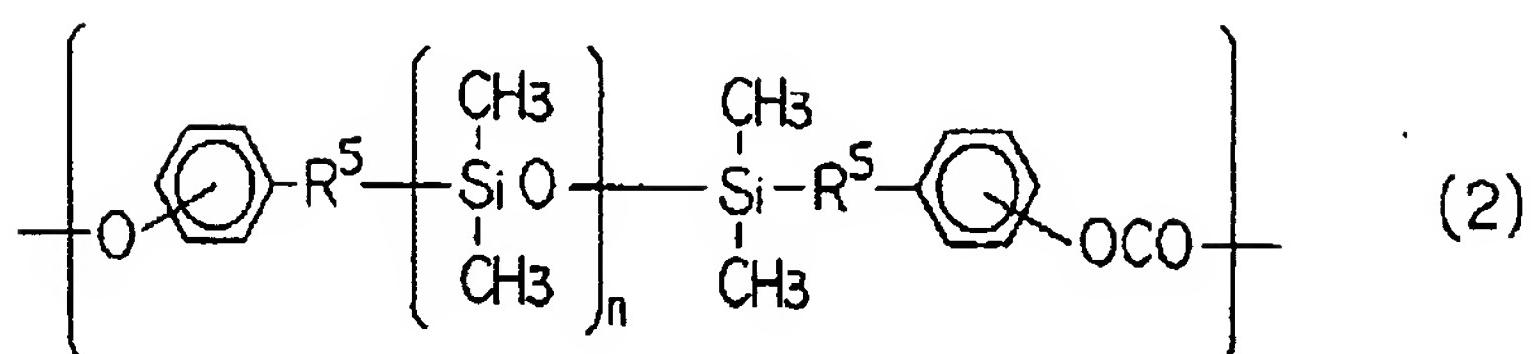
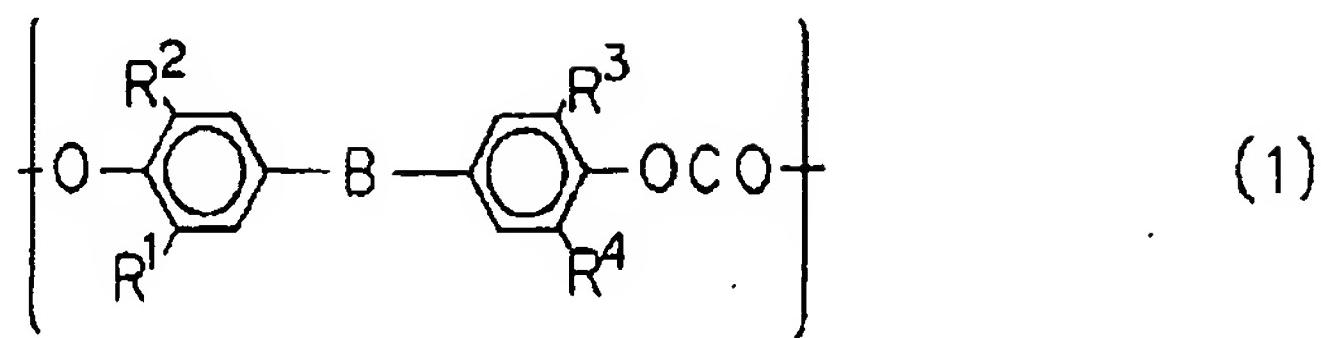
[Claim 2]

(B) [REDACTED]-[REDACTED] (1)
[REDACTED] (2) [REDACTED]
[REDACTED]-[REDACTED]
[REDACTED]

(B) polycarbonate-organopolysiloxane copolymer, is polycarbonate-organopolysiloxane copolymer which possesses structural unit which is displayed with below-mentioned Formula (1) and structural unit which is displayed with below-mentioned Formula (2) and polycarbonate resin composition* which is stated in Claim 1 which is made feature

10

[Chemical Formula 1]



(□(1)□□□□B □□□□ 1~10 □□□□□□□□□□□
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 -O-, -S-, -CO-□□□-SO₂-□□□R¹□R²□R³ □□ R⁴
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 □□n □ 1~200 □□□□□□□)

(In Formula (1), straight chain, branched chain of carbon number 1~10 or alkylidene group, aryl substituted alkylene group, allylene group, -O-, -S-, -CO- of cyclic or -SO₂- it shows B, alkylene group of carbon number 2~6 which carbon atom where R¹, R², R³ and R⁴, respectively, show alkyl group of the hydrogen, halogen or carbon number 1~4, in Formula (2), as for R⁵, aromatic group and direct bond have done has connected with hydrogen atom of at least one showing, n shows integer 1~200.)

[Claim 3]

It confronts polycarbonate resin 100 parts by weight, relative
parts by weight of phosphate ester-based compound and
therelative dparts by weight of polytetrafluoroethylene which
possesses fibril-forming ability, satisfybelow-mentioned
formula polycarbonate resin composition* which is stated in
Claim 1 or 2 which is made feature

0.

$$5 \leq cXd^2$$

Specification

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00001

A horizontal row of ten small, empty rectangular boxes, likely used for input fields or placeholder text in a form.

A decorative border at the bottom of the page featuring two rows of rectangular frames. Each frame contains a small square, creating a repeating pattern across the width of the border.

[Description of the Invention]

[00011]

[Technological Field of Invention]

this invention regards polycarbonate resin composition, furthermore details regard polycarbonate resin composition of flame resistance which is superior in mechanical*thermal

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property and external appearance.

[0002]

[Prior Art]

polycarbonate resin has had mechanical*thermal property which is superior, is widely utilized in industrially including automobile field, OA equipment field, electrical and electronics fields.

On one hand, multiple flame retardant development and investigation is done in order recently, OA equipment, household appliance product or other application demand of flame retardation of synthetic resin material which is used for center, is strong, to answer to these demands.

Usually, halogen compound etc is combined mainly in flame retardation of polycarbonate resin, furthermore antimony trioxide etc is jointly used is many as the flame retardant auxiliary agent.

But, when halogen compound is combined to synthetic resin as flame retardant, there is a possibility of causing environmental contamination at time of fire generation or time of incineration.

In addition, polycarbonate resin original mechanical property which is superior is impaired, color change when forming, furthermore, property decrease when you use for long period with high temperature and coloration or other problem has possessed.

Because of this quantity reduction of halogen compound which is used for flame resistance resin is desired.

[0003]

composition which combines aromatic oligomer phosphate ester to aromatic polycarbonate resin or other thermoplastic resin is disclosed in Japan Unexamined Patent Publication Showa 59-202240 disclosure, but in order to obtain flame resistance which is superior, it is necessary relatively to add aromatic oligomer phosphate ester to large amount, there is a problem that mechanical*thermal property is impaired.

In addition but, ABS type resin blending to polycarbonate resin, we to have done the attempt which it tries to improve fluidity etc, furthermore the attempt which combines phosphate ester-based compound for flame resistance improvement has done, as for polycarbonate resin composition which combines ABS type resin and phosphate ester-based compound, heat resistance being insufficient, There is a problem that use with application where high heat resistance is required is restricted.

[0004]

By fact that polycarbonate-organopolysiloxane copolymer is

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combined to polycarbonate resin, improvement method of the impact resistance, is disclosed in Japan Unexamined Patent Publication Showa55-160052 number and Japan Unexamined Patent Publication Showa61-215652disclosure etc, in additionalthough, in polycarbonate resin, polycarbonate blend which possesses siloxane copolymer and the phosphorus-containing compound is disclosed in Japan Unexamined Patent Publication Hei 7-150028disclosure, is superior in impact resistance, flame resistance, in the external appearance of molded article or point of stiffness it was not somethingwhich always it can be satisfied.

□0005□

[0005]

[Problems to be Solved by the Invention]

It is to offer polycarbonate resin composition where objective of this invention is superior in flame resistance, impact resistance, heat resistance, at same time is superior in molding external appearance and stiffness.

□0006□

[0006]

[Means to Solve the Problems]

As for this invention, being something which can be made in order to solve above-mentioned problem, gist consists of (A) polycarbonate resin 1~99 weight% and the (B) polycarbonate-organopolysiloxane copolymer 99~1 weight% and content of silicon combines (C) phosphate ester-based compound 0.1~5 parts by weight and the polytetrafluoroethylene 0.2~2 parts by weight which possesses (D) fibril-forming ability vis-a-vis polycarbonate resin 100 parts by weight under 0.5 weight%, exists in polycarbonate resin composition which becomes.

□0007□

[0007]

You explain in detail below, concerning this invention.

dihydric phenol and carbonate precursor reacting with solution method or melt method, it is something which is produced as (A) polycarbonate resin in this invention.

As dihydric phenol, 2 and 2 -bis (4 -hydroxyphenyl) propane [bisphenol A], you can list bis (4 -hydroxyphenyl) methane, 1,1-bis (4 -hydroxyphenyl) ethane, 2,2-bis (4 -hydroxy-3,5-dimethylphenyl) propane, 2,2-bis (4 -hydroxy-3-methylphenyl) propane, bis (4 -hydroxyphenyl) sulfide, bis (4 -hydroxyphenyl) sulfone, etc preferably bis (4 -hydroxyphenyl) alkane system, you can list those which designate especially bisphenol A as main raw material.

As carbonate precursor, you can list carbonyl halide, carbonyl ester or haloformate, etc can list the di haloformate and mixture of those of preferably phosgene, diphenyl carbonate, bivalent phenol.

When polycarbonate resin is produced, mixing alone or 2

□□□ 2 □□□□□□□□□□□□□□□□□□□□

kinds or more, it can also use the aforementioned dihydric phenol.

□0008□

2,6-□□□□-2,4,6-□□(4-□
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□~6 □□□□□

A horizontal row of 20 empty square boxes, intended for children to practice writing their names. The boxes are arranged in three rows: a top row of 10 boxes, a middle row of 10 boxes, and a bottom row of 2 boxes.

□0010□

(B) _____-_____

[0008] When polycarbonate resin is produced, it is possible also to make branched polycarbonate, furthermore making use of branching agent they are 0.01, - 3 mole %, preferably 0.1~1.0mole % vis-a-vis above-mentioned bivalent phenol compound as quantity of branching agent.

As branching agent, fluoroglycine, 2,6-dimethyl-2,4,6-tri (4 -hydroxyphenyl) heptene-3, 4,6-dimethyl-2,4,6-tri (4 -hydroxyphenyl) heptene-2, 1,3,5-tri (2 -hydroxyphenyl) benzol, 1,1,1-tri (4 -hydroxyphenyl) ethane, 2,6-bis (2 -hydroxy-5-methylbenzyl) - 4-methyl phenol, the;al, the;al ' and;al "-tri (4 -hydroxyphenyl) - polyhydroxy compound, and 3 and 3 -bis where it is illustrated with 1, 3 and 5 -tri isopropyl benzene etc (4 -hydroxy aryl) oxy indole (=isatin bisphenol), 5-chloro isatin, 5,7-dichlor isatin, 5-bromo isatin etc are illustrated.

[0009]

molecular weight of polycarbonate resin, with viscosity average molecular weight which is converted, with 10,000-100,000, is point more preferably 150,000~60,000 of moldability and molded article property from the solution viscosity which was measured with temperature 25 deg C making use of methylene chloride as the solvent.

When polycarbonate resin which has these viscosity average molecular weight is produced, it is possible also to add catalyst etc in order to promote suitable molecular weight regulator, reaction.

[00101]

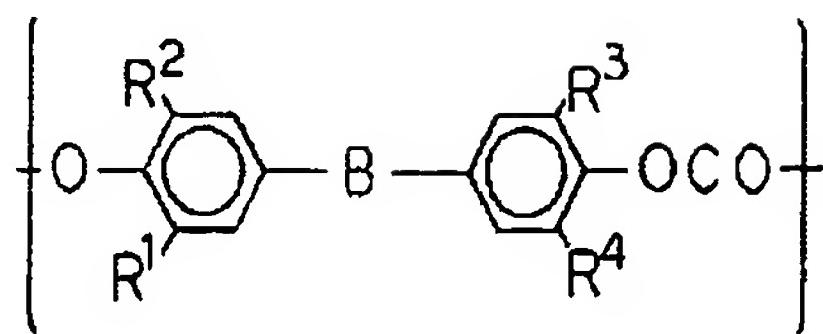
It is a copolymer which possesses block which was induced from the block and diorganosiloxane which were induced from aromatic polycarbonate as (B) polycarbonate-organopolysiloxane copolymer in this invention.

As polycarbonate-organopolysiloxane copolymer, it adjusts block which was induced from diorgano siloxane which consists of structural unit which is displayed with polycarbonate block and the Formula (2) which consist of structural unit to which for example Japan Unexamined Patent Publication Showa50-29695 number and the Japan Unexamined Patent Publication Hei 3-292359 number, copolymer which is disclosed in Japan Unexamined Patent Publication Hei 4-202466 number each disclosure is listed, is displayed with preferably, Formula (1) in molecule and it can list block copolymer which it possesses.

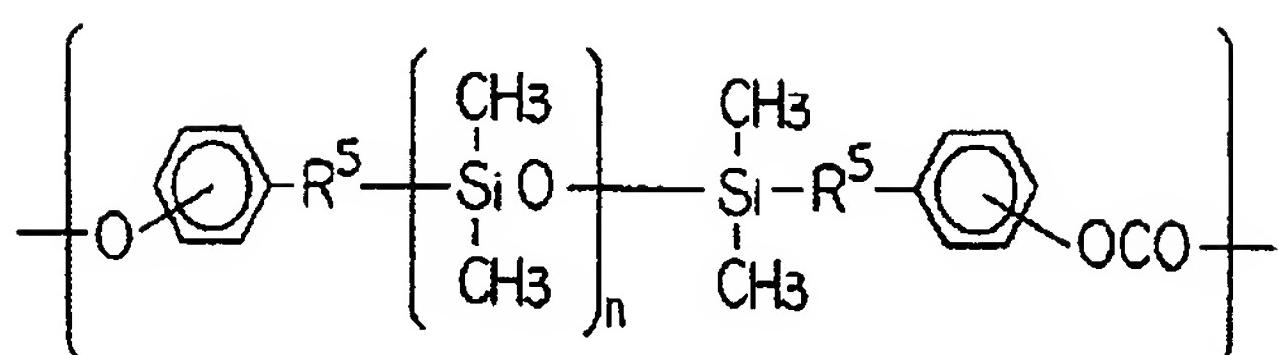
[00111]

□□ 2□

[Chemical Formula 2]



(1)



(2)

□0012□

□(1)□□□□B □□□□ 1~10 □□□□□□□□□□□□□
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□□□□□□□□-O-, -S-, -CO-□□□-SO₂-□□□□

R¹ R² R³ R⁴
1~4

□(2)□□□□R⁵ □□□□□□□□□□□□□□□□□□□
□□ 1 □□□□□□□□□□□□□□□□□□□ 2~6 □□□□□□□□
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(2) n 1-200

□□□□□□□□□□□□□□□□ n □□□□□ 5~100 □□□
□□□

□0013□

[0012]

In Formula (1), straight chain, branched chain of carbon number 1~10 or alkylidene group, aryl substituted alkylene group, aryl substituted alkylene group, allylene group, -O-, -S-, -CO- of cyclic or SO_2 - it shows B.

R^1 , R^2 , R^3 and R^4 , respectively, show alkyl group of hydrogen, halogen or carbon number 1~4.

In Formula (2), R⁵ aromatic group and shows alkylene group of carbon number 2~6 which carbon atom which direct bond has been done has connected with the hydrogen atom of at least one, concretely, can list ethylene, propylene, butylene, pentylene, hexylene or other group.

repeat number of siloxane unit in Formula (2) is integer 1 - 200

When n is under 1, improvement effect of impact resistance being insufficient, when it exceeds 200, fluidity is easy to decrease.

n is integer of preferably 5~100 from point of balance of the impact resistance and fluidity.

[0013]

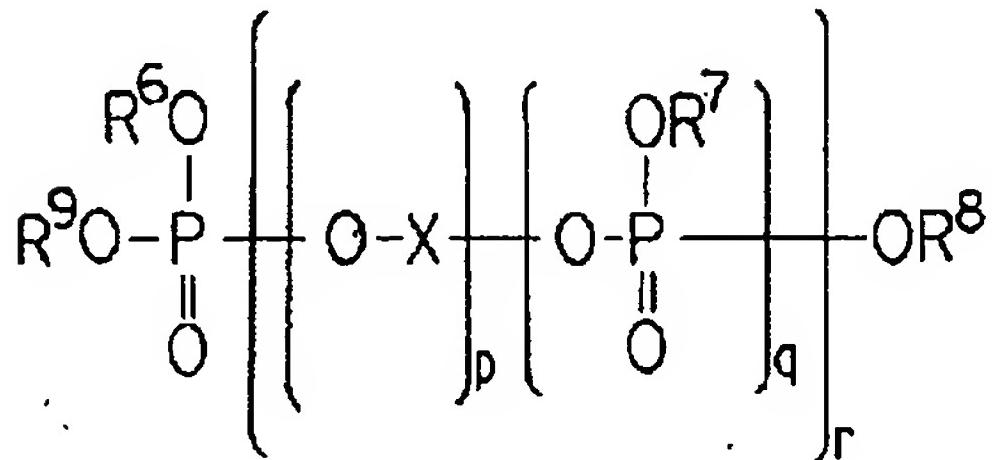
It is a preferably 2~40 weight%, more preferably 3~25 weight% as ratio of structural unit which is displayed with Formula (2) in polycarbonate-organopolysiloxane copolymer.

As polycarbonate-organopolysiloxane copolymer, produces

A horizontal row of 20 empty square boxes for writing responses.

□0018□

□□ 3□



□0019□

(3) R⁶ R⁷ R⁸ R⁹ H

□0020□

p,p' -

p □ 0 □□□ 1 □□□□q □ 1 □□□□□□□□□□□□ 1~30 □
□□□□□□r □ 0 □□□□□□□□□□□□ 1~10 □□□□□□

r 0 R⁶ R⁸ R⁹

with Formula (3) as (C) phosphate ester-based compound in this invention, but it is not something which is limited in these.

[0018]

[Chemical Formula 3]

(3)

[0019]

In Formula (3), R⁶, R⁷, R⁸ and R⁹, respectively, display the hydrogen atom or organic group, but R⁶, R⁷, R⁸ and R⁹ case where in eachcase is a H is excluded.

As organic group, you can list optionally substitutable, alkyl group, cycloalkyl group and aryl group etc.

When it is substituted, you can list for example alkyl group, alkoxy group, alkyl thio group, halogen, aryl group, aryloxy group, aryl thio group and halogenated aryl group etc as substituent, these substituent combined group (for example aryl alkoxy alkyl group etc) or you connect these substituent with oxygen atom, sulfur atom, nitrogen atom and etc are good even with combined group (for example aryl sulfonyl aryl group etc).

[0020]

X displays organic group of bivalent or greater, means basis of the bivalent or greater which it is possible organic group of bivalent or greater excluding one or more of hydrogen atom which from above-mentioned organic group has been connected to carbon atom can list group which is induced from for example alkylene group, (substituted) phenylene group, polynuclear phenol for example bisphenols, relative position of separation atomic valency of 2 or more is the option.

Especially, you can list hydroquinone, resorcinol, diphenylol methane, diphenylol dimethyl methane, dihydroxy biphenyl, p,p*-dihydroxy diphenylsulfone, dihydroxy naphthalene etc as desirable ones.

As for p with 0 or 1, as for q with integer of the integer, preferably 1~30 of 1 or more, as for r integer of integer, preferably 1~10 of 0 or greater is displayed.

However, when r_0 is, at least one inside $R^{⁶}$,

R⁸ and R⁹ displays organic group.

□0021□

[0021]

(□□□□□)□□□□□□□□(□□□□□□□)□□□□□
□□□(□□□□□□□)□□□□□□□□(2,3-□□□□□□□
□)-2,3-□□□□□□□□□□□□□□□□(2,3-□□□□□□□
□)□□□□□(□□□□□□□)□□□□□□□□□□□□□□R5~R8
□□□□□□□□□□□□□□□□□□□□□□□□□□□□□□□□
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As embodiment of phosphate ester-based compound, trimethyl phosphate, triethyl phosphate, tributyl phosphate, trioctyl phosphate, tributoxy ethyl phosphate, triphenyl phosphate, tricresyl phosphate, cresyl biphenyl phosphate, octyl biphenyl phosphate, diisopropyl phenyl phosphate, tris (chloroethyl) phosphate, tris (dichloropropyl) phosphate, tris (chloropropyl) phosphate, bis (2 and 3 -dibromopropyl) -2and 3 -dichloropropyl phosphate, tris (2 and 3 -dibromopropyl) and alkoxy for example methoxy, ethoxy and bisphenol A bis phosphate, hydroquinone bis phosphate, resorcinol bis phosphate, tri oxy benzene triphosphate etc of place where it is a propoxy, or preferably (substituted) phenoxy for example phenoxy, methyl (substituted) phenoxy you can list bis (chloropropyl) mono octyl phosphate, R5~R8,it is a preferably triphenyl phosphate and various bis phosphate.

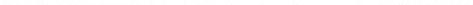
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It is good using these with alone and, 2 kinds or more combining, it is good using.

□0022□

[0022]

As ratio of phosphate ester-based compound, they are 0.1 - 5 parts by weight vis-a-vis (A) polycarbonate resin and polycarbonate resin100parts by weight which consists of (B) polycarbonate-organopolysiloxane copolymer.

0.1  5 


0.1 Under parts by weight flame resistance being insufficient, when it exceeds 5 parts by weight, heat resistance is easy to decrease.

From point of balance of flame resistance and heat resistance, ratio of the phosphate ester-based compound is preferably 0.5~4 parts by weight.

□0023□

[0023]

(D) ASTM 3

With ASTM standard classification it is done in type3 as polytetrafluoroethylene which possesses (D) fibril-forming ability in this invention, disperses in polymer easily, at same time connecting polymer, it is a compound which shows the tendency which makes fibrous material.

As polytetrafluoroethylene which possesses fibril-forming ability, we can be marketed from the for example Dupont-Mitsui Fluorochemicals Ltd., as Teflon6J or Teflon30J, or as Polyflon from Daikin Industries, Ltd., can procure easily.

□0024□

[0024]

They are 0.2 - 2 parts by weight vis-a-vis (A) polycarbonate resin and polycarbonate resin100parts by weight whichconsists of (B) polycarbonate-organopolysiloxane copolymer as ratio of polytetrafluoroethylene which possesses

the fibril-forming ability.

Ratio of polytetrafluoroethylene which possesses fibril-forming ability, under 0.2 parts by weight the melt dripping preventing effect when burning being insufficient, when it exceeds 2 parts by weight, external appearance is easy to become bad.

From point of balance of melt dripping preventing effect and external appearance, ratio of the polytetrafluoroethylene which possesses fibril-forming ability is preferably 0.3~1 part by weight.

[0025]

□0025□

Regarding to this invention, it confronts polycarbonate resin 100 parts by weight, relative cparts by weight of phosphate ester-based compound and relative dparts by weight of polytetrafluoroethylene which possesses the fibril-forming ability, below-mentioned formula is satisfied , is more desirablethan point of flame resistance, heat resistance, impact resistance.

[0026]

□0026□

10

[Mathematical Formula 1]

0.5□c×d□2

$$0.5 \leq cX_d^2$$

□0027□

[0027]

$c \times d = 0.5$  $c \times d = 2$ 

cX_d is easy to decrease under 0.5, flame resistance being insufficient, when cX_d exceeds 2, heat resistance and impact resistance.

□0028□

[0028]

A horizontal row of 20 empty square boxes, intended for handwriting practice of the uppercase letter 'V'. The boxes are arranged in a single line.

Mixing above-mentioned each component with mixer a tumbler, V type blender, Nauter mixer, Banbury mixer, kneading roll, extruder or other way, it can produce polycarbonate resin composition of this invention.

At time of producing polycarbonate resin composition of this invention, as for order of mixing method, mixture of each component there are not times when especially it is limited.

It makes preferred method and, it mixes all components beforehand with tumbler, V type blender etc, with extruder it is a method which melting and mixing is done in the uniform, but either using method which mixes remaining to blend of 2 kinds or more in these component according to geometry of component does not become inconvenient.

□0029□

[0029]

A grid of 40 empty square boxes arranged in four rows of ten. The grid is used for labeling or identifying individual components in a larger diagram.

In range which does not impair effect of this invention, flame retardant etc of various additive, for example stabilizer, mold release, ultraviolet absorber, dye and pigment, inorganic filler, other public knowledge of quantity which that effect reveals can be contained in polycarbonate resin composition of

(6)

ASTM D-790

kg/cm²

□0032□

A horizontal row of 20 small square icons, followed by two additional small square icons below them.

(9) **SiPC-2** 5%
1.75% 22.000

(11)□□□□□□□□-2(□□□□□□□□-2 □□□
□□)□□□□□□□□□□□□□□□□□□□□□□□□□□□□□□ TPP□

(13)ABS □□(□□ ABS □□□□□)□□□□□□□(□)□□
□□□□□□□ ET 70□

□0033□

□□□□□□□□□□ 120 deg C □ 6 □□□□□□□□□□
(□□□□□□□□□□ J-50EP) □□□□□□□□□□ 270

(6) flexural modulus

Following to ASTM standard D-790, it executed bending test of ASTMD-790 making use of flexural test piece which it drew up.

It indicated flexural modulus, with unit of kg/cm².

[0032]

In each Working Example or Comparative Example below, raw material which is used is below-mentioned sort.

(7) polycarbonate resin (Below PC it states.), Mitsubishi Engineering Plastics Corp. (DB 69-164-9669) make, tradename IupilonS-3000, viscosity average molecular weight22,000*

(8) polysiloxane-polycarbonate copolymer-1 (Below SiPC-1 it states.), polycarbonate-organopolysiloxane copolymer where quantity of poly dimethylsiloxane is 15 weight% of copolymer entirety (As for silicon content 5.25 weight%), viscosity average molecular weight22,000*

(9) polysiloxane-polycarbonate copolymer-2 (Below SiPC-2 it states.), polycarbonate-organopolysiloxane copolymer where quantity of poly dimethylsiloxane is 5 weight% of copolymer entirety (As for silicon content 1.75 weight%), viscosity average molecular weight 22,000*

(10) phosphate ester-based compound-1 (Below phosphate ester-1 it states.), condensed phosphate ester, Daihachi Chemical Industry Co. Ltd. (DN 69-072-8662) make, tradenamePX-200*

(11) phosphate ester-based compound-2 (Below phosphate ester-2 it states.), triphenyl phosphate, Daihachi Chemical Industry Co. Ltd. (DN 69-072-8662) make, tradename TPP*

polytetrafluoroethylene (Below PTFE it states.)
polytetrafluoroethylene, Daikin Industries Ltd. which
possesses (12) fibril-forming ability (DB 69-054-0356) make,
tradename PolyflonF201L*

(13) ABS resin (Below ABS it states.), Mitsui Toatsu Chemicals Inc. (DB 69-053-6982) make, tradename SantacET-70*

[00331]

After mixing each component which is stated in { Working Example1~5 and Comparative Example1~5 } Table 1, Table 2, with the proportion of same listed, 40 mm diameter single screw extruder (Isuzu Kako Co., Ltd. make) were used, extrusion it did with cylinder temperature 250 deg C and pelletizing did.

With cylinder temperature 270 deg C, die temperature 80 deg C, designated test piece it formed pellet which it acquires, 6

hours after drying, making use of injection molding machine (The Japan Steel Works Ltd. (DB 69-056-8472) Ltd. make, tradename J-50EP) with 120 deg C.

□□□□□□□□□□□□□□□□-1 □□□□-2 □□□□

composition and property evaluation result of pellet, were shown in Table 1 and Table 2.

□0034□

[0034]

10

[Table 1]

表-1

	実施例-1	実施例-2	実施例-3	実施例-4	実施例-5
(A)PC	82	94	94	94	92
(B)SiPC-1		6	6	6	8
SiPC-2	18				
(C)リン酸エステル-1	2	2		0.5	2
リン酸エステル-2			2		
(D)PTFE	0.5	0.5	0.5	2	0.5
燃焼性	V-0	V-0	V-0	V-0	V-0
燃焼時間	20	20	20	32	18
荷重たわみ温度	129	129	126	134	120
衝撃強度	74	74	70	78	72
外観	◎	◎	◎	◎	○
曲げ弾性率	23,000	23,000	23,000	22,400	22,600

□0035□

[0035]

□□ 2□

[Table 2]

表-2

	比較-1	比較-2	比較-3	比較-4	比較-5
(A)PC	100	100	94	94	67
(B)SiPC-1			6	6	33
(C)リン酸エスチル-1	2	2	0.05	10	2.5
(D)PTFE	0.5	0.5	3	0.04	0.04
(E)ABS		3			
燃焼性	V-0	V-1	V-1	V-2	V-0
燃焼時間	28	52	56	37	28
荷重たわみ温度	130	129	135	108	118
衝撃強度	11	19	79	5	78
外観	◎	◎	×	◎	×
曲げ弹性率	23,900	23,600	21,900	26,300	19,100

□0036□

[0036]

卷之三

[Effects of the Invention]

polycarbonate resin composition of this invention is superior in flame resistance, impact resistance, stiffness, heat resistance, at same time weare superior even in external appearance, it is very useful as material of the electronic and electric appliances, OAequipment or other housing or sash

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application, various part.